Jun Pan, Guo-Kai Jia, Fang Liu, Xiao-Ming Chen* and Xing-Yu Zhang*

# Crystal structure of 4-methoxyphenyl-3-phenylpropiolate, $C_{16}H_{12}O_3$

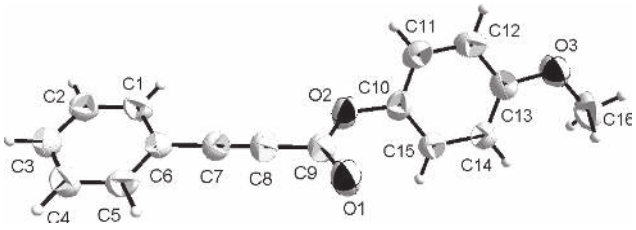


**Figure 1:** The structure of the title compound showing 40% probability displacement ellipsoids and the atom-numbering scheme.

https://doi.org/10.1515/ncrs-2019-0279
Received April 17, 2019; accepted June 20, 2019; available online September 9, 2019

## Abstract

$C_{16}H_{12}O_3$, orthorhombic, *Pbca* (no. 19), $a = 3.9935(16)$ Å, $b = 16.629(7)$ Å, $c = 19.406(8)$ Å, $V = 1288.7(9)$ Å$^3$, $Z = 4$, $R_{gt}(F) = 0.0387$, $wR_{ref}(F^2) = 0.1084$, $T = 296(2)$ K.

**CCDC no.:** 1935386

The molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

## Source of material

A 25 mL Schlenk tube was charged with phenylpropiolic acid (1.0 mmol), *p*-methoxyphenol (1.2 mmol), dimethylaminopyridine (0.5 eq), dicyclohexylcarbodiimide (1.5 eq) in 10 mL of dichloromethane step by step. The reaction mixture was stirred at room temperature for 24 h. Upon the reaction completion (monitored by TLC), the reaction mixture was filtered, quenched with 5 mL of water, extracted with EtOAc (5 mL), washed with brine. The combined organic

***Corresponding authors: Xiao-Ming Chen,** Department of Biology and Chemistry, Hunan University of Science and Engineering, Yongzhou, Hunan 425199, China; and **Xing-Yu Zhang,** South China University of Technology, Guangzhou, Guangdong 510641, China, e-mail: zhangxingyu_good@126.com (X.-Y. Zhang)
**Jun Pan, Guo-Kai Jia and Fang Liu:** Department of Biology and Chemistry, Hunan University of Science and Engineering, Yongzhou, Hunan 425199, China

**Table 1:** Data collection and handling.

| | |
|---|---|
| Crystal: | Colourless block |
| Size: | 0.32 × 0.28 × 0.24 mm |
| Wavelength: | Mo $K\alpha$ radiation (0.71073 Å) |
| $\mu$: | 0.09 mm$^{-1}$ |
| Diffractometer, scan mode: | Bruker APEX-II, $\varphi$ and $\omega$ |
| $\theta_{max}$, completeness: | 27.5°, >99% |
| $N(hkl)_{measured}$, $N(hkl)_{unique}$, $R_{int}$: | 14445, 2930, 0.046 |
| Criterion for $I_{obs}$, $N(hkl)_{gt}$: | I > 2s(I), 2342 |
| $N(param)_{refined}$: | 173 |
| Programs: | Bruker [1], SHELX [2] |

**Table 2:** Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å$^2$).

| Atom | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| O1 | 0.0581(5) | 0.45567(9) | 0.71069(8) | 0.0831(6) |
| O2 | 0.3362(5) | 0.47637(9) | 0.61209(7) | 0.0693(5) |
| O3 | 0.3527(4) | 0.80804(9) | 0.61734(8) | 0.0717(5) |
| C1 | 0.2742(6) | 0.16495(12) | 0.54743(10) | 0.0564(5) |
| H1 | 0.364233 | 0.200526 | 0.515431 | 0.068* |
| C2 | 0.2484(6) | 0.08498(13) | 0.53169(11) | 0.0654(6) |
| H2 | 0.321996 | 0.066373 | 0.489106 | 0.078* |
| C3 | 0.1146(7) | 0.03219(14) | 0.57838(12) | 0.0695(7) |
| H3 | 0.094768 | −0.021968 | 0.567150 | 0.083* |
| C4 | 0.0102(6) | 0.05892(13) | 0.64145(12) | 0.0681(6) |
| H4 | −0.075234 | 0.022672 | 0.673434 | 0.082* |
| C5 | 0.0310(5) | 0.13927(13) | 0.65771(10) | 0.0585(5) |
| H5 | −0.045932 | 0.157453 | 0.700178 | 0.070* |
| C6 | 0.1665(5) | 0.19318(11) | 0.61096(9) | 0.0492(4) |
| C7 | 0.1872(6) | 0.27691(12) | 0.62753(10) | 0.0569(5) |
| C8 | 0.1999(6) | 0.34677(12) | 0.64120(11) | 0.0641(6) |
| C9 | 0.1870(6) | 0.43072(12) | 0.65996(10) | 0.0582(5) |
| C10 | 0.3325(6) | 0.56091(12) | 0.61879(10) | 0.0546(5) |
| C11 | 0.2015(6) | 0.60363(14) | 0.56481(10) | 0.0600(5) |
| H6 | 0.107468 | 0.576865 | 0.527453 | 0.072* |
| C12 | 0.2098(6) | 0.68617(13) | 0.56611(10) | 0.0609(5) |
| H7 | 0.118952 | 0.715420 | 0.529794 | 0.073* |
| C13 | 0.3528(5) | 0.72575(12) | 0.62125(10) | 0.0521(5) |
| C14 | 0.4847(6) | 0.68230(12) | 0.67511(10) | 0.0549(5) |
| H8 | 0.580232 | 0.708897 | 0.712397 | 0.066* |
| C15 | 0.4755(6) | 0.59918(12) | 0.67399(10) | 0.0555(5) |
| H9 | 0.565233 | 0.569610 | 0.710236 | 0.067* |
| C16 | 0.5085(7) | 0.85106(13) | 0.67193(14) | 0.0819(7) |
| H10 | 0.499909 | 0.907662 | 0.662337 | 0.123* |
| H11 | 0.737765 | 0.834378 | 0.676002 | 0.123* |
| H12 | 0.392982 | 0.840201 | 0.714300 | 0.123* |

layers were dried over anhydrous $Na_2SO_4$, filtered, concentrated *in vacuo* and the residue was purified by chromatography on silica gel, using Hexane/EtOAc (10:1) as the eluent, give the 4-methoxyphenyl 3-phenylpropiolate as a colorless solid. The solid was dissolved in ethylene acetate and crystals of the title compound were obtained by slow evaporation within a week.

## Experimental details

Hydrogen atoms were placed in their geometrically idealized positions and constrained to ride on their parent atoms.

## Comment

In recent decades, thousands of alkynyl-containing active ingredients have been isolated from traditional medicinal plants or other active organisms [3, 4]. These alkynyl-containing active ingredients exhibit abundant biological activities, such as anti-tumor [5], anti-inflammatory [6], antibacterial, anti-viral, etc [7, 8]. Acetylenic acid ester compounds as reactants in organic synthesis, in the core skeleton structure of conjugated structures has an important position [9]. Therefore, it is important to find a highly efficient and simple synthetic protokol of conjugated olefin derivatives for the supplement of active compounds [10].

There is one molecule in the asymmetric unit of the title structure (see the figure). The single crystal structure verifies that all bond lengths are in normal ranges and in accord with parameters reported in the literature [11, 12].

**Acknowledgements:** The work was supported by the opening project of key laboratory of comprehensive utilization of advantage plants resources in Hunan south, Hunan University of Science and Engineering (XNZW16C07), The plant of Yongzhou Science and Technology innovation guidance program (2018ZD16), Research Programs of Hunan University of Science and Engineering (17XKY015).

## References

1. Bruker. APEX2, SAINT and SADABS. Bruker AXS Inc., Madison, WI, USA (2009).
2. Sheldrick, G. M.: SHELXT-integrated space-group and crystal-structure determination. Acta Crystallogr. **C71** (2015) 3–8.
3. Aline, B.; Annie, L.; Arnaud, H.: Alkynylation of chiral aldehydes: alkoxy-, amino-, and thio-substituted aldehydes. Chem. Rev. **106** (2006) 2355–2403.
4. Alanko, J.; Kurahashi, Y.; Yoshimoto, T.: Panaxynol, apolyacetylene compound isolated from oriental medicines, inhibits mammalianlipoxygenases. Biochem. Pharmacol. **48** (1994) 1979–1981.
5. Metzger, B. T.; Barnes, D. M.; Reed, J. D.: Purple Carrot (Daucus carota L.) polyacetylenes decrease lipopolysaccharide-induced expression of inflammatory proteins in macrophage and endothelial cells. J. Agric. Food Chem. **56** (2008) 3554–3560.
6. Resch, M.; Heilmann, J.; Steigel, A.: Further phenols and polyacetylenes from the rhizomes of Atractylodes lancea and their anti-inflammatory activity. Planta Med. **67** (2001) 437–439.
7. Yang, M. C.; Kwon, H. C.; Kim, Y.-J.; Lee, K. R.; Yang, H. O.: Oploxynes A and B, polyacetylenes from the stems of Oplopanax elatus. J. Nat. Prod. **73** (2010) 801–805.
8. Hong, S. S.; Lee, J. H.; Jeong, W.; Kim, N.; Jin, H. Z.; Hwang, B. Y.; Lee, H.-J.; Lee, S.-J.; Jang, D. S.; Lee, D.: Acetylenic acid analogues from the edible mushroom Chanterelle (Cantharellus cibarius) and their effects on the gene expression of peroxisome proliferator-activated receptor-gamma target genes. Bioorg. Med. Chem. Lett. **22** (2012) 2347–2349.
9. Zhang, S. L.; Deng, Z. Q.: Synthesis of quinolines and naphthyridines *via* catalytic retro-aldol reaction of β–hydroxyketones with ortho-aminobenzaldehydes or nicotinaldehydes. Org. Biomol. Chem. **14** (2016) 8966–8970.
10. Chen, X. M.; Zhang, X. Y.; He, F. L.; Pan, J.; Jia, G.-K.: Crystal structure of (2*E*,4*Z*)-dimethyl-4-((phenylamino)methylene)pent-2-enedioate, $C_{14}H_{15}O_4N$. Z. Kristallogr. NCS **234** (2019) 87–89.
11. D'Souza, D. M.; Kiel, A.; Herten, D.-P.; Rominger, F.; Muller, T. J. J.: Synthesis, structure and emission properties of spirocyclic benzofuranones and dihydroindolones: a domino insertion–coupling–isomerization–diels–alder approach to rigid fluorophores. Chem. Eur. J. **14** (2008) 529–547.
12. Lellek, V.; Hansen, H.-J.: Unexpected thermal transformation of aryl 3-arylprop-2-ynoates: formation of 3-(diarylmethylidene)-2,3-dihydrofuran-2-ones. Helv. Chim. Acta **84** (2001) 3548–3580.